Synthesis and Characterization of $BiPbSr_2XO_6$ (X=Mn, Fe)

A thesis submitted in partial fulfilment for the award of degree in

Master of Science
In
Physics

By

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Academic year: 2012-2014

DECLARATION

I hereby declare that the work carried out me at Department of Physics, National Institute of Technology, Rourkela. I further declare that to best of my knowledge the carried out experimental work has not formed the basis for the award of any degree, diploma, or similar title of any university or institution.

Date:

Place: Rourkela

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CERTIFICATE

This is to certify that the thesis entitled "**Synthesis and Characterization of $BiPbSr_2XO_6$ (X=Mn, Fe)**" being submitted by **JNANESWAR KHETI** in partial fulfilment of the requirements for the award of the degree of Master of Science in Physics at National Institute of Technology, Rourkela is an authentic experimental work carried out by her under our supervision. To the best of our knowledge, the experimental matter embodied in the thesis has not been submitted to any other University/Institute for the award of any degree or diploma.

Date:

Dr. Anil K. Singh

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I am very thankful **to Dr. S. K. Sarangi, Director**, for giving me the opportunity to do this project. My heartfelt thanks to **Prof. D.K. Bisoyi** sir, Head of the department. I wish to express my sincere thanks to our guide **Prof. Anil K. Singh** for his continues encouragement and supervision on this project work. Without his guidance I would have never reached to the completion.

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ABSTRACT

$BiPbSr_2MnO_6$ is a potential candidate where we can search for possible magnetoelectricity near 200K. The compound is prepared by solid state route reaction using bismuth oxide (Bi_2O_3), lead oxide (PbO), strontium carbonate ($SrCO_3$) and manganese oxide (Mn_2O_3).We could not get clean phase of $BiPbSr_2MnO_6$ after several attempts. Then, we prepare the compound $BiPbSr_2FeO_6$, by replacing Mn ions by Fe. The XRD analysis conform the formation of the single phase compound and the lattices constant are nearly equal to the calculated one. The FESEM image shows the compound is well sintered; grains are distributed uniformly and less porosity present. From the UV-VIS plot the band gap is found to be 1.57 eV. Dielectric constant (ε_r) and loss ($\tan\delta$) as function of frequency at room temperature shows contribution of space charge and orientational polarization.

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INTRODUCTION

1.1 Magnetism

Magnetism is physical property of magnet that attracts or repels when another magnet is come closer. The materials which show magnetism in presence or absence of external magnetic field are called as magnetic materials. We can classify magnetic materials on the basis of susceptibility and domain orientation into five categories such as diamagnetic, paramagnetic, ferromagnetic, anti-ferromagnetism and ferrimagnetism.

(a) Diamagnetism

These types of materials show negative susceptibility, which means they repel the external magnetic field applied on them. The repulsion is caused by diamagnetic substance is due to the tiny current flow by the electrons in opposite direction to the magnetic field in accordance with Lenz's law. Diamagnetic substance reduces the density of lines of forces when placed in a magnetic field. Antimony, bismuth, and mercury, gold and copper are the examples of this magnet.

(b) Para magnetism

For paramagnetic substance susceptibility is small and positive. Para magnetism is due to the orientations of permanent magnetic dipoles due to the presence of all external magnetic fields. Susceptibility varies inversely with the temperature in accordance with Curie law

$$\chi = C/T$$

where C is Curie constant. Manganese, platinum, tungsten belongs to this category.

(c) Ferromagnetism

Here the susceptibility is positive and high. Ferromagnetic materials exhibit magnetic properties even in the absence magnetic field. Magnetization is increased when an external magnetic field is applied. The relation between susceptibility of Ferro magnetic substance and temperature is given by Curie-Weiss law

$$\chi = C / (T-T_c)$$

where T_c is critical temperature or transition temperature above which ferromagnetic substance changes to paramagnetic substance. Examples of ferromagnetic substances are iron, cobalt and nickel.

(d) Anti ferromagnetism

The susceptibility is positive but small. In anti-ferromagnetic materials the neighbouring magnetic moments are anti-parallel to each other. So the net magnetic moment cancel out and the materials are behaves as paramagnetic materials. These types of materials also show par magnetism above Neel temperature. The temperature dependence of this type of susceptibility for these types of materials is

$$\chi = C / (T-\theta)$$

where θ is Neel temperature or transition temperature. Chromium, nickel oxide (NiO) etc. shows antiferro magnetic behaviour below Neel temperature.

(e) Ferrimagnetisms

Ferrimagnetism materials have more complex structure. Ferri magnetic substances have dipoles alignment similar to that of anti-ferromagnetic substance but magnitudes are different.

Examples: YIG (yttrium iron garnet), $PbFe_{12}O_9$ and $BaFe_{12}O_9$ etc.

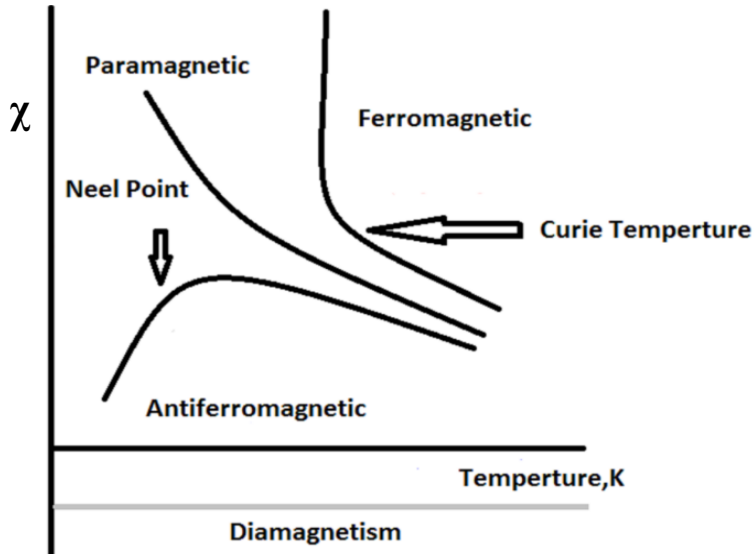


Fig1.1: Susceptibility as a function of temperature for different magnetic materials

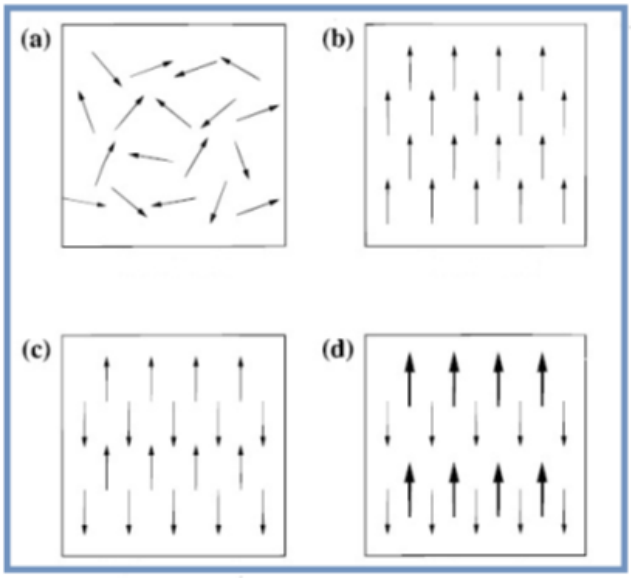


Fig. 1.2: The spin arrangement of (a) paramagnetic (b) ferromagnetic (c) antiferromagnetic and (d) ferrimagnetic material

1.2 FERROELECTRIC

The ferroelectric effect is an electrical phenomenon where crystals are exhibit a spontaneous dipole moment by applying external field.Ferroelectric materials have domains and show a hysteresis response to polarization and electric field. They have potential applications as capacitors because of their concentration of electric flux density and used in electrochemical transducer and actuators. Examples of ferroelectric materials are Rochelle salt and perovskite oxides.

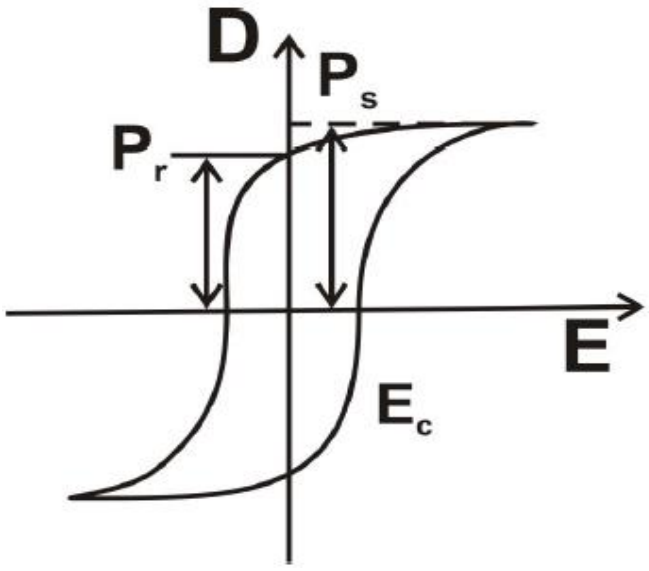


Fig 1.3: Hysteresis loop of a ferroelectric material

A typical hysteresis loop is shown in the figure above. Below and above the coercive field a ferroelectric material behaves like an ordinary dielectric but at E_c polarization reverse occurs and the dielectric shows non linearity. The area under the curve is the amount of energy

required to twice reverse the polarization. At zero electric field the electric displacement within a single domain can have two values corresponding to the opposite orientations of the spontaneous polarization. Multi-domain crystal gives two extremes at zero electric field. In principle the spontaneous polarization is equal to the saturation value of the electric displacement extrapolated to zero fields. The remnant polarization P_r is different from spontaneous polarization P_s when reverse nucleation occurs before the applied field reverses. This is due to the presence of internal (or external) stress or if the free charges below the surfaces cannot reach their equilibrium distribution during each half-cycle of the loop.

1.3 FERROELASTICITY

Materials which show spontaneous strain are called Ferro elastic materials. In ferroelastic materials when a stress is applied to it, materials undergo a phase change with different crystal structure or different orientation by inducing spontaneous strain. Nickel titanium is an example of ferroelastic material.

1.4 MULTIFERROIC

Multiferroic means existence of two or more ferroic properties in a single phase. Multiferroics, the term introduced by H. Schimd (in 1994), materials that exhibit two or more primary ferroic properties (ferroic order parameter) simultaneously in same phase. The four ferroic properties are given as

1. Ferromagnetism
2. Ferroelectricity
3. Ferroelasticity
4. Ferrotoroidicity

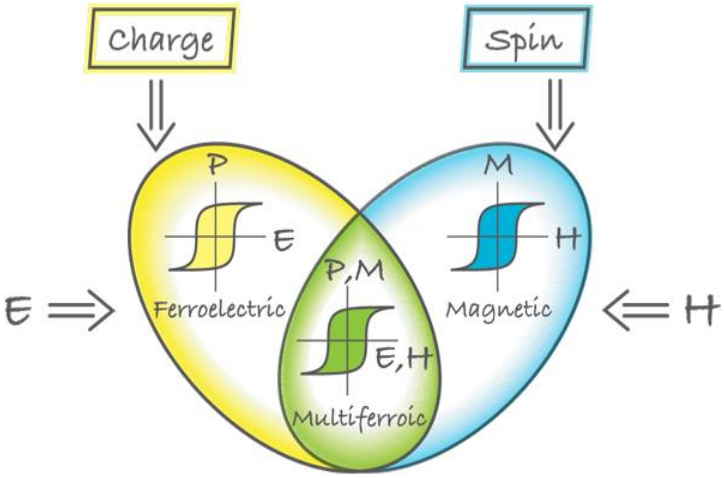


Fig 1.4: Blue circle displays the hysteresis of ferromagnetic materials, yellow circle displays usual hysteresis of ferroelectric materials and the circle in between these two indicates hysteresis of multiferroics where coupling occurs.

[D. Khomskii, Phys. 77, 50937 (2009)]

Typical examples of multiferroics are given below

- Perovskite transition metal oxides (e.g. $TbMnO_3$)
- Rare earth manganites with chemical composition $ReMnO_3$ (Re^{3+}= Lu, Ho, Y)
- Ferrites ($LuFe_2O_4$)
- Bismuth compound ($BiFeO_3$ and $BiMnO_3$)
- Non-oxides such as ($BaNiF_4$)
- Spinel chalcogenides, e.g. $ZnCr_2Se_4$

Symmetry and multiferroicity

On the basis of symmetry operation the ferroic orders can be subdivided in two categories: space inversion symmetry operation and time inversion symmetry operations. Ferroelectric, ferromagnetic, ferroelastic and ferrotorrodic order parameters show space inversion and time inversion symmetry as shown below in the table:

	Space invariant	Space variant
Time invariant	Ferro elastic	Ferroelectric
Time variant	Ferromagnetic	Ferrotoroidic

So to show magnetoelectric multiferroic both space and time inversion should be violated.

Mechanics for ferroelectricity in multiferroics

Basing on the origin of ferroelectricity in multiferroic material system, they can be distinguishing as **proper** and **improper** ferroelectrics.

I. Proper ferroelectrics:

Ferroelectric distortion is the primary order parameter in proper ferroelectricity. $BaTiO_3$ is an example of this type of ferroelectricity, where a covalent bonding between the transition metal and the oxygen happens to allow a polar state.

a) Lone pair multiferroics

In perovskite crystals the ferroelectric distortion arises due to the B site shift towards the oxygen octahedral. Transition metal need empty d-shell to show the ferroelectric property where as to show magnetism property partial filled d-shell is required. So to show multiferroic property A cation gives the lone pair to B site to made it partially filled d-orbit, which gives magnetism property.$BiFeO_3$, $BiMnO_3$ and $PbVO_3$ are example of this type of multiferroic.

II. Improper ferroelectrics:

Phase change is the order parameter for improper ferroelectricity. The FE bend is a secondary order parameter in the sense that it is compelled by the existence of other order parameters. When inversion symmetry of the crystal is broken by magnetic spiral, this types of ferroelectrics comes to picture.

(a)Charge ordering

Charge ordering is one of the reasons for the multiferroic property. Compounds having magnetic frustration and ion of mixed valence have such order. Due to polar arrengment in this type of compound improper ferroelectricity is coming. $LuFe_2O_4$ is the best example of this type of multiferroic.

b) Magnetically driven ferroelectricity

Magnetically determined multiferroics are insulating materials, typically oxides, in which macroscopic electric polarization is made by magnetic long-range order. An important but not suitable form for the appearance of spontaneous electric polarization is the absence of inversion symmetry. When the inversion symmetry of a crystal is broken by magnetic structure like spiral magnetic ordering.

1.5 TYPES OF MULTIFERROICS

Depending on the microscopic origin of multiferroicity, these classes of compounds are divided in two major categories:

I. Type I multiferroic

II. Type II multiferroic

(I)Type- I Multiferroics

In these types of materials electricity and magnetism have different source and they appear independent of one another. So there is a coupling between them. In these materials coupling between ferroelectricity and magnetism is weak. In these materials ferroelectricity occurs at higher temperature than magnetism. Ferroelectric and magnetic transition occurs at above room temperature. Polarization is very high of the order of 10-100 $\mu C/cm^2$. There are four different subclasses of type-I multiferroics:

(a) Multiferroic Perovskites

Perovskite magnetic materials having ferroelectricity belongs to this type of multiferroic. Perovskites like $BaTiO_3$ is ferroelectric. Magnetism needs partially filled d shells of a transition metal, but all ferroelectric perovskites have empty d shell. The covelent bond between the transition metal and oxygen due to the off centre shift of transition metal cause the ferroelectric property in the compound. d electrons of magnetic transition metals preventing ferroelectricity in magnetic perovskites. This is called as "d^0 vs d^n" problem. Solution to this problem is by making mixed perovskites with d^0 and d^n ions.

(b) Ferroelectricity due to lone pair

In $BiFeO_3$, $BiMnO_3$ and $PbVO_3$, Bi^{3+} and Pb^{3+} plays important role in the origin of ferroelectricity. Electrons which don't take part in chemical bonding they are called lone pairs. In these ions, there are two outer 6s electrons. They have high polarizability as the condition required for ferroelectricity.

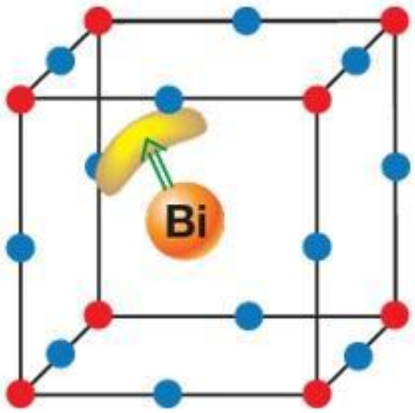


Fig.1.5: Ordering of lone pairs Bi^{3+} and Pb^{2+} ions

(c) Ferroelectricity due to charge ordering:

Here inequivelent sites have different charges, so that inequivelent bond develops ferroelectricity. In charge ordering process both sites and bonds turn out to be inequvalent, which creates ferroelectricity.

(d) Geometric ferroelectricity:

Here tilting of off centre sites develops ferroelectricity. In $YMnO_3$ the MnO_5 block tilts around the magnetic Mn, which is at centre. Tilting is happening to provide packed structure. So oxygen moves toward Y atom because of orientation. The Y-O bonds give dipoles and ferroelectricity develops.

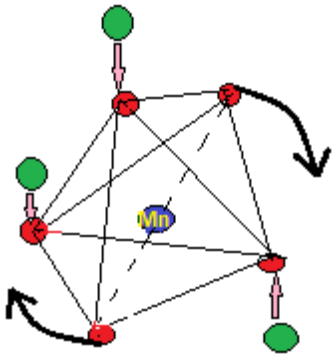


Fig.1.6: Generation of polarization due to tilting of MnO_5 polyhedra in $YMnO_3$

(ii) Type –II Multiferroics

In these types of materials magnetism causes ferroelectricity implying a strong coupling between them. In these multiferroics, magnetoelectric coupling is very strong. Polarization is usually very small compared to type-I multiferroics; it is of the order 10-2 $\mu C/cm^2$. There are two subclasses of type-II multiferroics

(a)Spiral type-II multiferroics:

Most of the type II multiferroics belongs to this subgroup. Ferroelectricity appears with a spiralling magnetic phase, mostly of cycloid type. Magnetic frustration is a source of spiral magnetic ordering in insulators. Frustrated system gives these types of multiferroic.

(b) Collinear magnetic structures:

In this type of multiferroic ferroelectric property is driven due to the collinear magnetic structure. So, that all magnetic moments are aligned along a particular axis without involvement of the spin orbit interaction. Polarization in these materials can appear as a consequence of exchange interactions because the magnetic coupling varies with the atomic positions. At high temperature the distance between the ions along the chains are the same, the chain has inversion symmetry. Magnetic ordering breaks the inversion symmetry, the spins forms an antiferromagnetic structure. Due to exchange interaction the distortion of ferromagnetic and antiferromagnetic bonds are different.

1.6 WHY THERE ARE FEW MULTIFERROICS?

Due to the less existence of ferroelectric and ferromagnetic materials in nature, the chance of existence of multiferroics materials is also very less. Multiferroic materials were developed since 1960, but due to the insufficient laboratory facilities the numbers are less. The main reasons for existence of few numbers of multiferroics are

(a) Symmetry:

There are 31 point groups allows polarization and there are 31 point group allows magnetization. But there are only 13 point group which allows both the polarization and magnetization. This represents a considerable reduction of multiferroics materials. Therefore from symmetric point of view less number of multiferroics materials exists.

(b) Electrical properties:

Ferroelectric materials are insulator and ferromagnetic materials are conductor. So we assume that the lack of the simultaneous occurrence of both ferromagnetic and ferroelectric property. There are also very few antiferromagnetic ferroelectrics, even though antiferromagnets are usually insulating materials.

(c) d^0-ness:

Magnetism occurs due to partially filled d-orbit, the tendency for it to make a distortion that removes the centre of symmetry is eliminated. But ferroelectricity occurs due to empty

d-orbit. This could be the result of a number of effects, including size, more dominant distortion and electronic properties.

1.7 Applications

Multiferroic materials are used in high sensitive magnetic field sensor, filter, oscillator etc.

One can use in data storage system, where in both magnetic and electric state of the compound can store the information. Multiferroic thin film can also use in magnetoelectric device.

2. MOTIVATION BEHIND PRESENT WORK:

$BiPbSr_2MnO_6$ is a potential candidate where we can search for possible magnetoelectricity near 200 k. microscopic origin of multiferroicity in $BiPbSr_2MnO_6$ has not revealed yet. There is a structural ambiguity in $BiPbSr_2MnO_6$ that conformed from XRD and Neutron diffraction study. We can prepare compound like $BiPbSr_2FeO_6$ and $BiPbSr_2CoO_6$ by replacing Mn and Co to search multiferroicity and its microscopic origin in these compounds.

3.1 CRYSTAL STRUCTURE OF $BiPbSr_2MnO_6$ and $BiPbSr_2FeO_6$:

The crystal structure of $BiPbSr_2MnO_6$ is orthorhombic with lattice parameter a=5.3311 Å, b=5.3988 Å, c=23.757 Å with space group Amaa. The crystal structure of $BiPbSr_2FeO_6$ is also orthorhombic with lattice parameter a=5.42 Å, b=5.49 Å, c=23.20 Å and it is belongs to Fmmm space group.

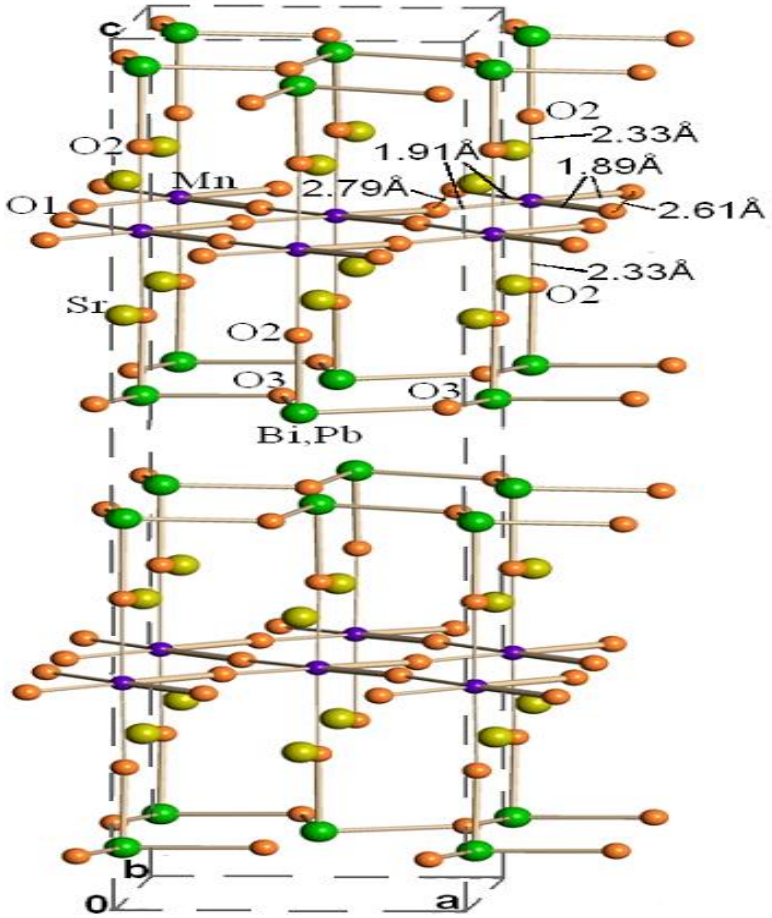


Fig 3.1: **Structure of $BiPbSr_2MnO_6$**

From x-ray diffraction techniques the crystal structure of $BiPbSr_2MnO_6$ is nonecentersymmetry and from powder neutron diffraction techniques the structure is cenetrsymmetry. Though the structure is diffrent in XRD and neutron diffraction techniques, the atomic parameters are similar in both the cases. The main diffrence is in the centrosymmetry the oxygen atoms in the (Bi,Pb)O layer along a axis are disorder in two position. The octahedra MnO_6 are coupled by common vertices O1 into perovskite-type layers in the ab plane. But in the noncentrosymmetry the Mn ion is displaced from the octahedron center along the a axis. Bi^{3+} and Pb^{+2} ions equally share same position in both the structure. With two nearest oxygen ions, one of which serves as the axial vertex of the MnO_6 octahedron, they form tetrahedra $(Bi,Pb)O_3E$, thus attaching to the manganese octahedral layers from both sides. The stereochemically active lone electron pairs of Bi^{3+} and Pb^{2+} do not polarize the structure, since their directions are different: they are directed to the space between formed sandwich-like layers, thus separating them from each other.

3.2 MAGNETOELECTRIC PROPERTIES

From the magnetization curve i.e. susceptibility vs. temperature the magnetoelectric temperature for $BiPbSr_2MnO_6$ is 190 k.

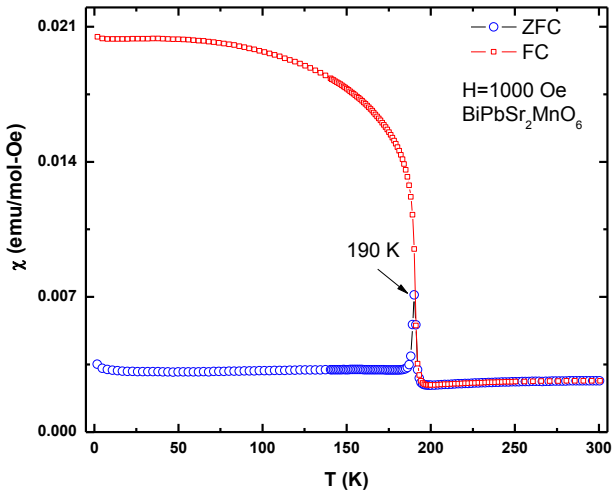


Fig 3.2: **Susceptibility vs temperature graph for $BiPbSr_2MnO_6$**

The magnetic transition temperature for $BiPbSr_2FeO_6$ is 240 K and magnetoelectric temperature is 90 K.

4 Preparation of samples

4.1 Synthesis of $BiPbSr_2MnO_6$:

- For the preparation of 2gm of $BiPbSr_2MnO_6$ through solid state reaction route by using 0.6013 gm of Bi_2O_3 , 0.6013 gm of PbO, 0.7954 gm of $SrCO_3$ and 0.2126 gm of Mn_2O_3.
- It follows the reaction

$$½\ Bi_2O_3 + PbO + 2SrCO_3 + ½\ Mn_2O_3 \rightarrow BiPbSr_2MnO_6$$

Calculation:

The atomic weight of Bi = 208.98040 amu

Pb = 207.2 amu

Sr = 87.62 amu

Mn = 54.938045 amu

O = 15.999 amu

C = 12.011 amu

Molecular weight of Bi_2O_3 = 2(208.98040) + 3(15.999)

= 465.9578 amu

$SrCO_3$ = 87.62 + 12.011 + 3(15.999) amu

= 147.628 amu

$PbO = 207.2 + 15.999$ amu

$= 223.199$ amu

$Mn_2O_3 = 2(54.938045) + 3(15.99)$ amu

$= 157.87309$ amu

$BiPbSr_2MnO_6 = 208.98040 + 207.2 + 2\times (87.6) + 54.938045 + 6\times (15.999)$

$=742.352445$ amu

For 2 gm of $BiPbSr_2MnO_6$ required weight of

½ Bi_2O_3 = 465.9578 / 742.352445 = 0.62767 gm

PbO = (223.199 × 2) / 742.352445 = 0.60132 gm

$2SrCO_3$ = (295.256 × 4) / 742.352445 = 0.79546 gm

½ Mn_2O_3 = 78.936545/ 742.352445 = 0.2126659528 gm

PRESSING

All the samples were ground in silica mortar pestle for two hours in order to maintain homogeneity. The powders were pressed using stainless die of diameter 10 mm. The pressure was applied to the powder sample using Hydrolytic press of pressure ~ 6 ton. The thicknesses of the final sintered pallets were ~ 1mm.

SINTERING

The pellets are kept in the furnace and sintered at 700^0 c for 15 hours. The ramping rate of temperature is 4°C/min. After sintering the pallets were again grinded for one hour and pellets are prepared by pressing. In the second sintering the pellets are kept at 700^0 c for 15 hours. For the 3^{rd} sintering the same process followed as done in 2^{nd} sintering. After 3^{rd} sintering XRD is done for the sample.

4.2 Synthesis of $BiPbSr_2FeO_6$:

- For the preparation of 2gm of $BiPbSr_2FeO_6$ through solid state reaction route by using .626911 gm of Bi_2O_3 , 0.60059 gm of PbO , 0.7944 gm of $SrCO_3$ and 0.2148 gm of Fe_2O_3.
- It follows the reaction

$$½\ Bi_2O_3 + PbO + 2SrCO_3 + ½\ Fe_2O_3 \rightarrow BiPbSr_2FeO_6$$

Calculation:

The atomic weight of Bi = 208.98040 amu

Pb = 207.2 amu

Sr = 87.62 amu

Fe = 55.845 amu

O = 15.999 amu

C = 12.011 amu

Molecular weight of Bi_2O_3 = 2× (208.98040) + 3× (15.999) amu

= 465.9578 amu

$SrCO_3$ = 87.62 + 12.011 + 3× (15.999) amu

= 147.628 amu

PbO = 207.2 + 15.999 amu

= 223.199 amu

Fe_2O_3 = 2× (55.845) + 3× (15.99) amu

= 159.687 amu

$BiPbSr_2FeO_6$ = 208.98040 + 207.2 + 2× (87.6) + 55.845 + 6× (15.999)

= 743.2594 amu

For 2 gm of $BiPbSr_2FeO_6$ required weight of

½ Bi_2O_3 = 465.9578 / 743.2594 = 0.62691gm

PbO = (223.199 × 2) / 743.2594 = 0.60059 gm

2$SrCO_3$ = (295.256 * 4) / 743.2594 = 0.794489 gm

½ Fe_2O_3 = 79.8435 / 743.2594 = 0.214846 gm

PRESSING

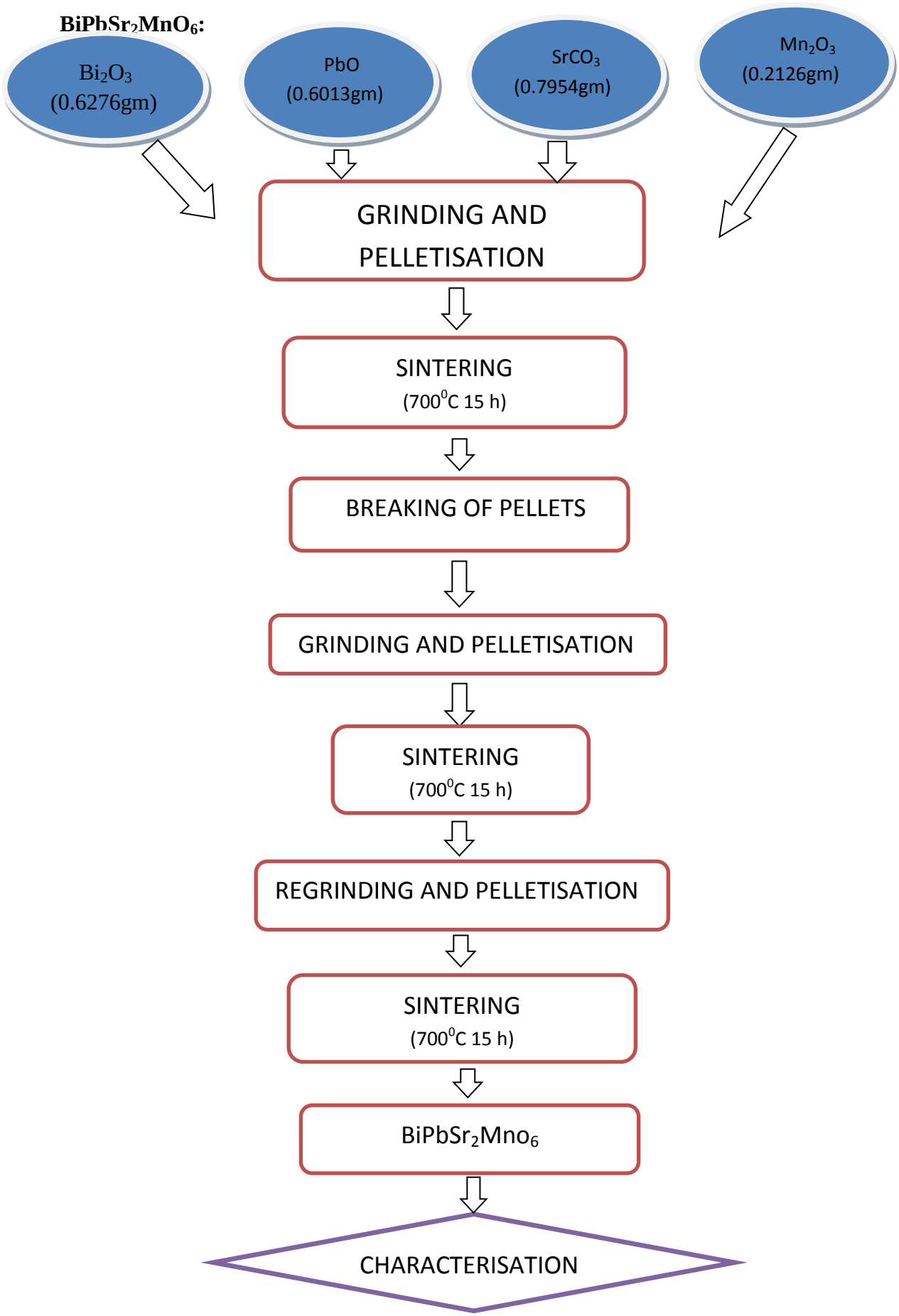
All the samples were ground in silica mortar pestle for two hours in order to maintain homogeneity. The powders were pressed using stainless die of diameter 10mm. The pressure was applied to the powder sample using Hydrolytic press of pressure ~ 6 ton. The thicknesses of the final sintered pallets were ~ 1mm.

SINTERING

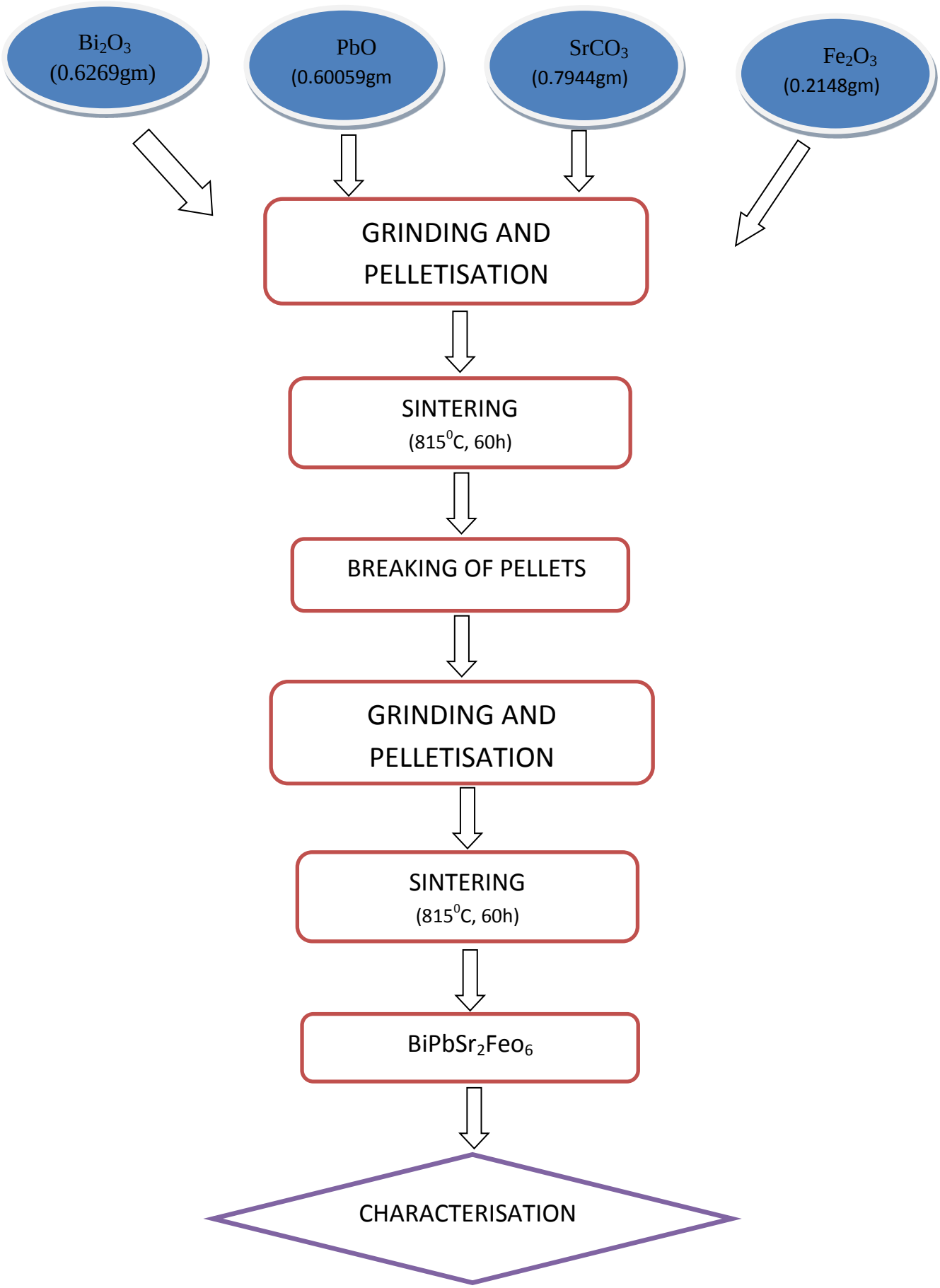
The pellets are kept in the tubular furnace at 815^0 C for 60 hours. After sintering the pellets are grinded for 1 hour and pellets are prepared by pressing. The pellets are again kept at 815^0 C for 60 hours. After that samples are characterized by XRD, FESEM, UV-VIS, Dielectric measurement for its study.

FLOW CHART FOR STEP FOLLOWED FOR SAMPLE PREPARATION:

$BiPbSr_2MnO_6$:



$BiPbSr_2FeO_6$:



6 CHARACTERIZATION TECHNIQUES:

6.1 XRD –:

X-ray diffraction technique is a nondestructive technique to study the structure of crystal by using Bragg's law. We can consider crystal structure consist of planes or layers which behaves as semitransparent mirrors. When X-ray having wavelength similar to that of the inter planner distance, incident in such a way that the angle of reflection is equal to the angle of incidence and diffraction occurs. It can be explained by Bragg's law.

According to Bragg's law

$$2d \sin\theta = n\lambda$$

Where d = inter planner distance

θ = angle between incident ray and scattering surface

n = an integer

λ = wavelength of the incident radiation

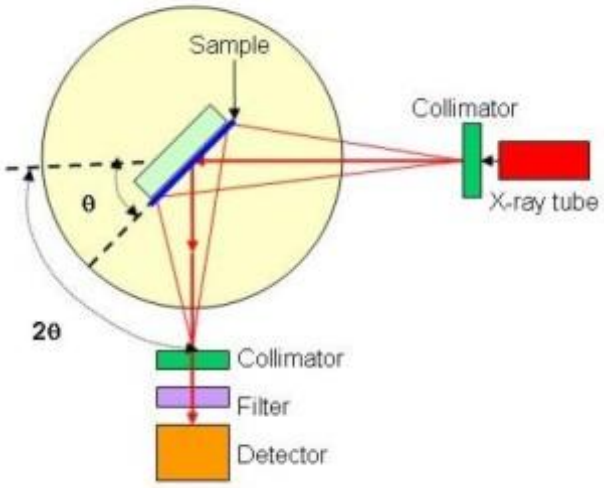


Fig 6.1: Schematic diagram of XRD

Characteristic x-rays are produced by using x-ray tube. Matched filters are used in XRD instrument to check out the continuum radiation. The apertures in a strong x-ray absorber collimate the x-rays, which results narrow x-ray beam. These narrow beams are allowed to strike on the sample surface. To satisfy the Bragg's condition for diffraction, the spectrometer arrangement couples the rotation of the crystal with the rotation of the detector

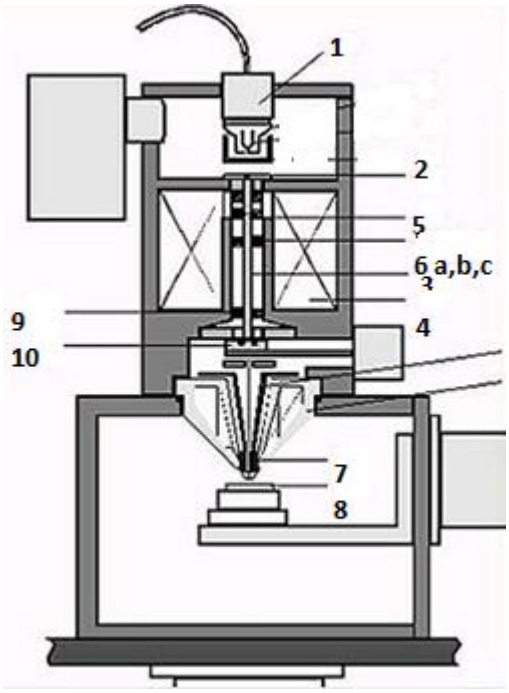
to make the angle of rotation of the detector is twice that of the crystal of the x-rays from the crystal lattice planes.

6.2 FESEM AND EDAX:

The field effect scanning electron microscope (FESEM) is a non-destructive and versatile technique for the morphological study and composition of the sample. We can get high contrast, less electrically distorted images from the FESEM. Semiconductor device cross sectional analysis for gate width, gate oxide and thin film thickness can also extract from FESEM.

FUNCTION:

To perform FESEM, samples are first gold coated. Then samples are mounted on the sample holder. Field emission source emits high energetic electron in presence of high electric field gradient, which are called as primary electrons. Narrow beams of primary electrons are produced by focussing these primary electrons by using electronic lens, which deflect them and bombarded on the sample. This bombardment results in production and ejection of secondary electrons. The surface structure of the sample affects the angle and velocity of secondary electrons. These secondary electrons are detected by the detector to produce electrical signal. After amplification the signals are transformed to a video scan image, which can be seen on the screen.



1- Field emission gun
2- Anode
3- Vacuum tube
4- Lens
5- Apertures
6- Alignment coils
7- Deflecting system
8- Objective lens
9- Stigmator
10- Isolating valves

Fig 6.2: Schematic diagram of FESEM

EDX:

This is the electron dispersive X-Ray spectroscopy. Elemental & compositional anlysis of a sample can be perfromed by using this technique. Here an high energetic electron beam is bombarded on the sample.this bombardment result in ejection of an inner shell electron. To fill this vacancy a higher energy level electron will be jump down by radiating x-rays. All the elements have different x-rays wavelengths. So by detecting these x-rays by a x-ray detector, we can detect the elements present in the sample.

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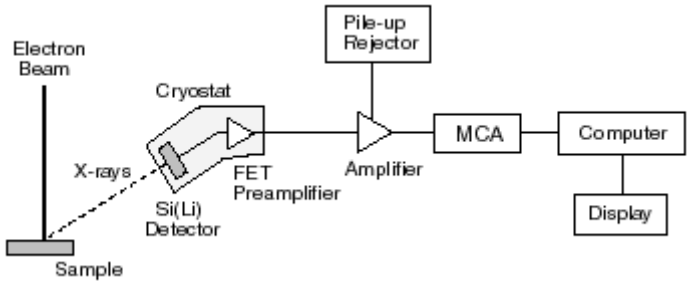

Fig 6.3: Schematic diagram of EDS

6.3 UV-VIS SPECTROSCOPY:

It is a type of absorption spectroscopy or reflectance spectroscopy. It occurs in the ultraviolet-visible spectral region that is it uses light in the visible and its adjacent ranges. The colours of the chemicals involved in the synthesis of the sample are affected by the absorption or reflectance in the visible range. In this technique, absorption measures electronic transitions of molecules in this region of electromagnetic radiation from the ground state to the excited state.

The basic principle of this technique is how the molecule absorbs energy in the form of electromagnetic radiation to excite the electron to higher states. The ease of excitation of the electrons affects the absorption of light by the sample. Radiations of different wavelengths are absorbed by different molecules.. Its range lies between 160-780 nm.

In order to calculate the band gap of a sample we have used the following formula

$$(\alpha h\nu)^2 = B(h\nu - E_g)$$

where B : a constant related to a transition probability

E_g : Band gap

n : an index characterizing the transition step

n=2 for indirect band gap

n= ½ for direct band gap

6.4 DIELECTRIC SPECTROSCOPIC ANALYSIS:

This spectroscopic analysis method helps to measures the dielectric properties of a medium as a function of frequency. Its basic principle is the interaction of external field with the electric dipole moment of the sample which is described by permittivity. Dielectric constant is the measure of the polarisibility of a material that is its strong opposing nature towards the movement of polar molecule. Dielectric loss is the measure of dissipation of electromagnetic energy of a dielectric material in the form of heat. Permittivity can be expressed as a complex quantity with a real and an imaginary part.

$\varepsilon = \varepsilon' - j\varepsilon''$

Where ε= permittivity of the medium

ε'= real part of permittivity

ε"=imaginary part of permittivity

Tangent loss is given by $\tan\delta = \varepsilon'' / \varepsilon'$

7 RESULTS AND DISCUSSION-

7.1 X-RAY ANALYSIS:

In the X-ray diffraction, the sample is scanned in the range 5^0 to 80^0 at step rate 3^0 per second. The XRD plot is given below. The prominent peaks in the plot are indexed to various [hkl] planes using XPERT high score.

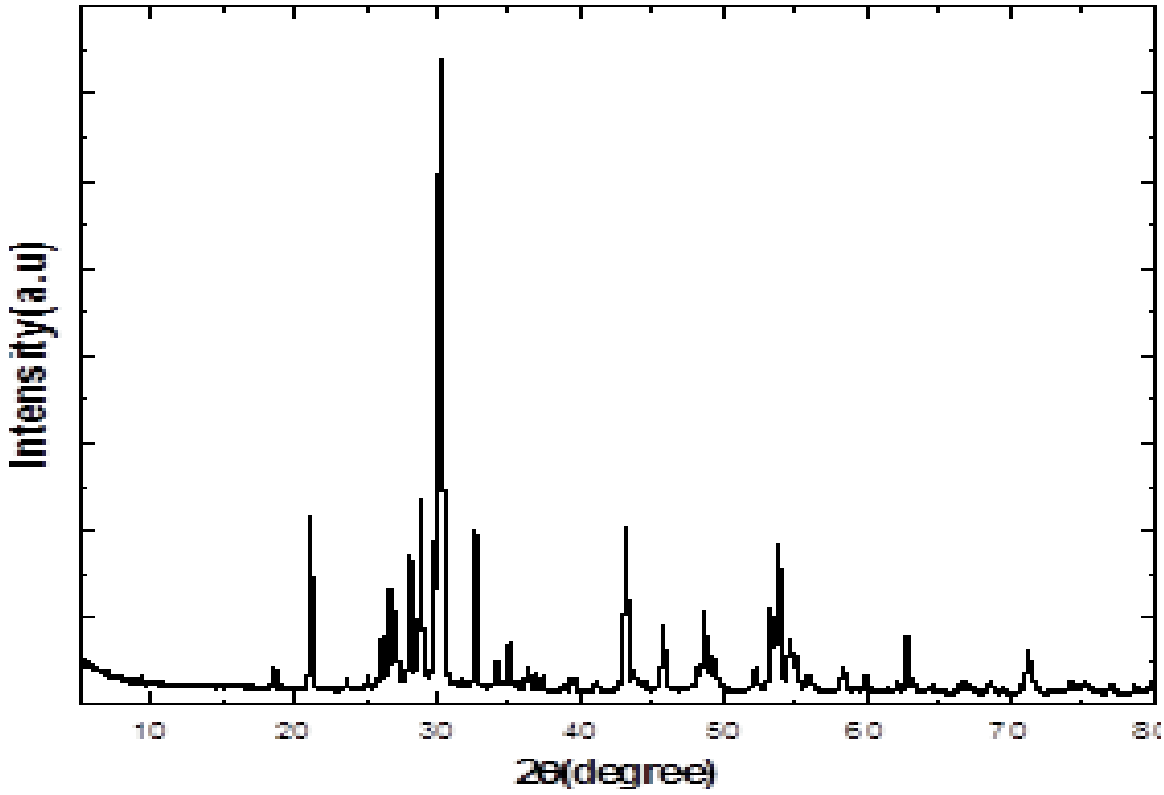

Fig7.1: XRD PLOT OF $BiPbSr_2MnO_6$

As from the XRD plot of $BiPbSr_2MnO_6$ the compound is not in single phase.

The theoretical values of lattice parameters of $BiPbSr_2FeO_6$ a, b and c are 5.42 Å, 5.49 Å and 23.22 Å respectively. Here I have calculated the lattice parameters by using the formula for orthorhombic crystal system as $1/d^2 = h^2/a^2 + k^2/b^2 + l^2/c^2$ by taking the planes (002), (020),(200), (008) and the values of a, b and c are found as 5.39 Å, 5.48 Å and 23.03 Å respectively.

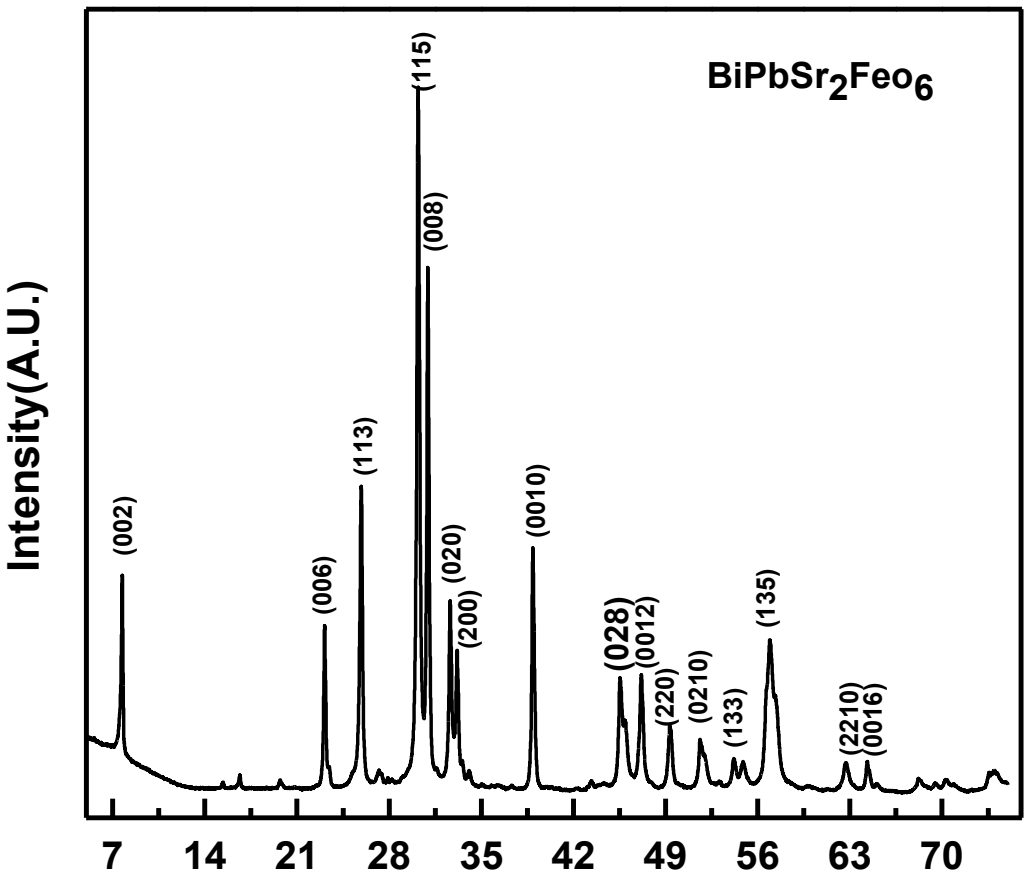

Fig7.2: XRD Plot of $BiPbSr_2FeO_6$

7.2 FESEM AND EXD ANALYSIS:

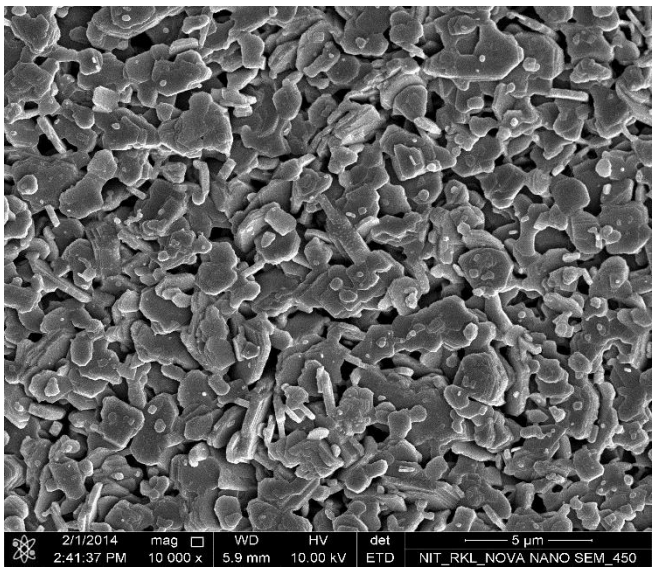

Fig7.3: FESEM IMAGE AT 5 µm

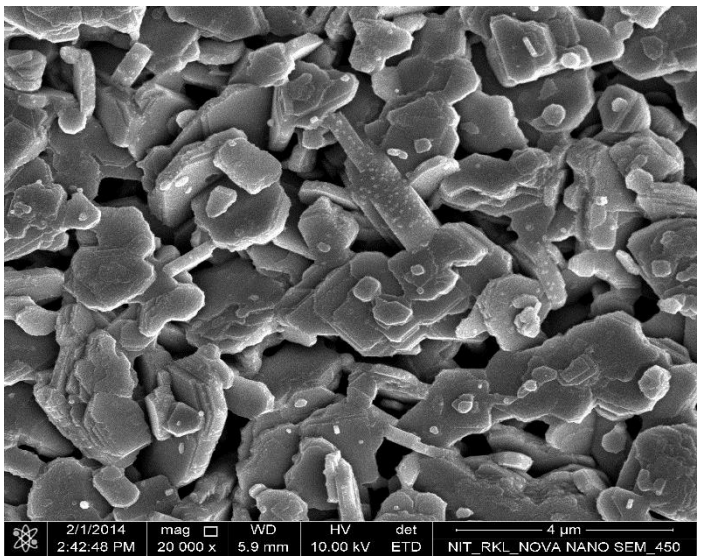

Fig7.4: FESEM IMAGE AT 4 µm

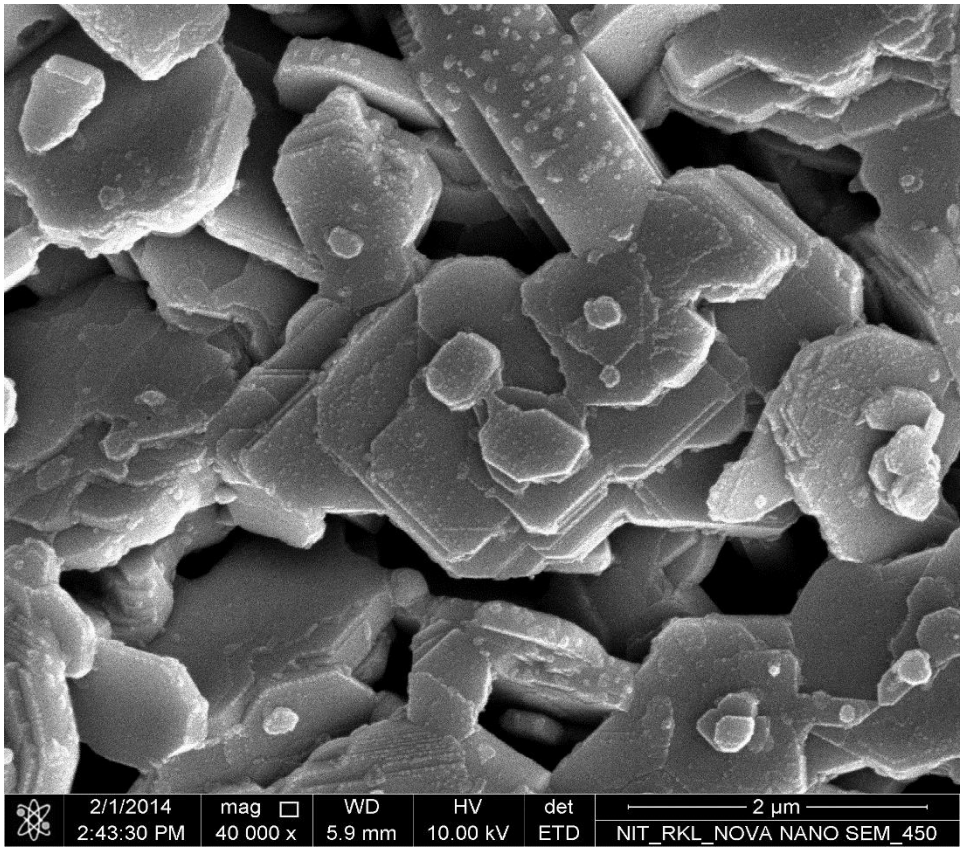

Fig7.5: FESEM IMAGE AT 2 µm

From the FESEM data of $BiPbSr_2FeO_6$ the distribution of grain is uniform and also the porosity of the sample is less. Also the compound is well sintered. The grain size also found as 3.154 µm.

EXD

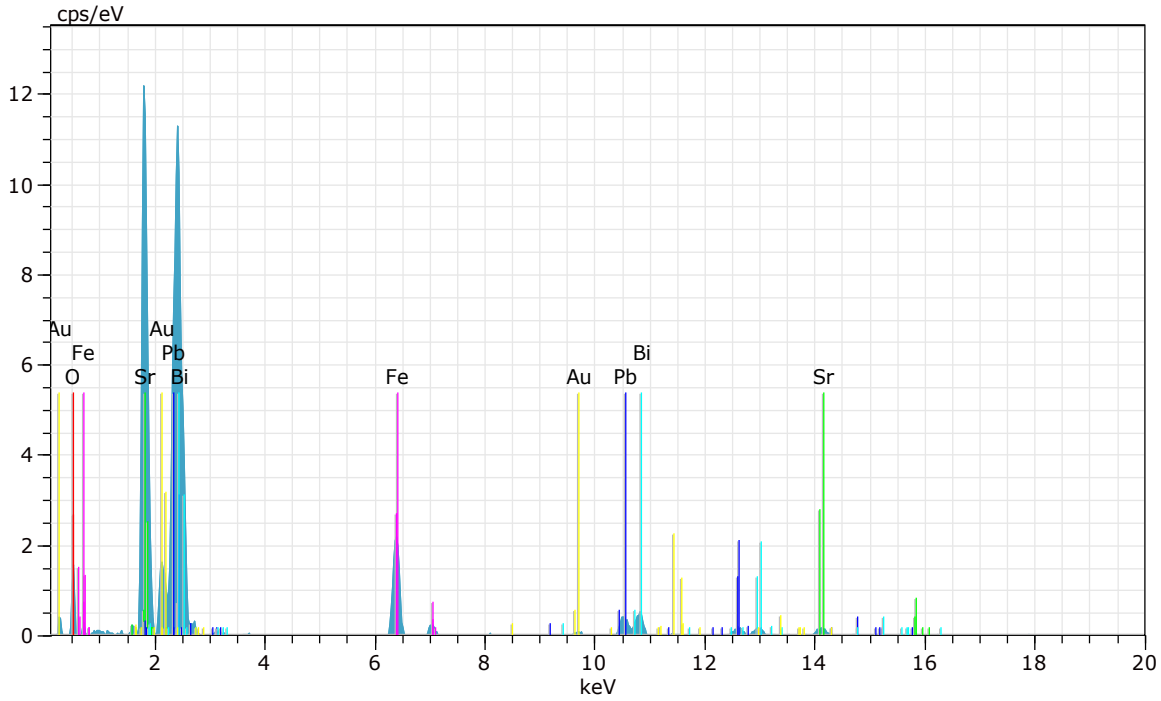

Fig7.6: EDX SPECTRUM OF $BiPbSr_2FeO_6$

Spectrum: spectrum 11.spx

El	AN	Series	Net unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Sr	38	L-series	31285	19.99	33.72	30.32	0.86
Bi	83	L-series	2962	16.46	27.76	10.47	0.64
Pb	82	L-series	2696	12.37	20.86	7.93	0.49
Fe	26	K-series	7604	6.01	10.14	14.30	0.20
O	8	K-series	2227	4.45	7.51	36.99	0.85
Au	79	L-series	380	0.00	0.00	0.00	0.00

From the EDX results the composition of $BiPbSr_2FeO_6$ is conformed. The gold is coming because of the pellet is gold coated before the FESEM characterisation. From the spectrum data the weight of different elements are same as the theoretical values.

7.3 UV-VIS ANALYSIS:

In the figure given below is the graph between wavelength vs absorbance and energy vs $(\alpha h\nu)^2$. From the graph between energy vs $(\alpha h\nu)^2$ I have calculated the band gap for the $BiPbSr_2FeO_6$, which is equals to 1.57 eV. This is near to the semiconductor range.

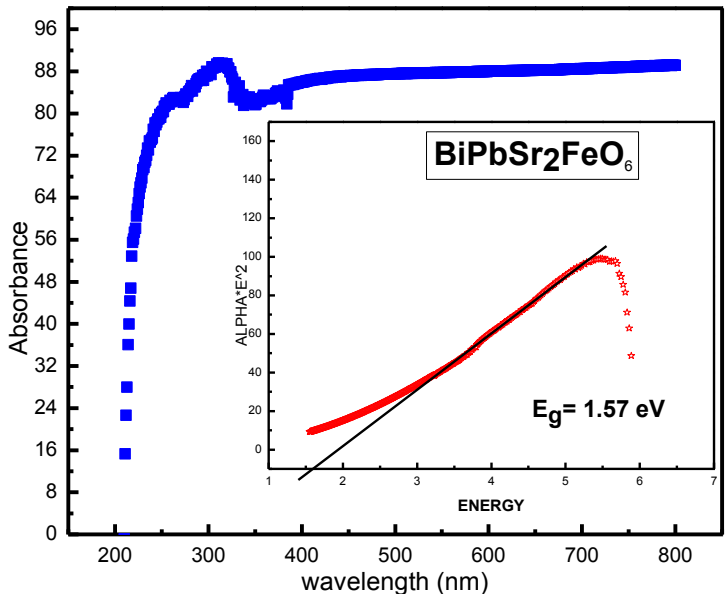

Fig7.7: Absorption spectra and energy band gap for $BiPbSr_2FeO_6$

7.4 DIELECTRIC ANALYSIS:

The dielectric measurement as a function of frequency in the range 100 Hz to 1 MHz is done shows the variation of tan δ with the frequency. We can see from the figure 7.8 that tan δ decreases linearly with frequency while dielectric constant is decreases with frequency and become constant after 1 kHz.

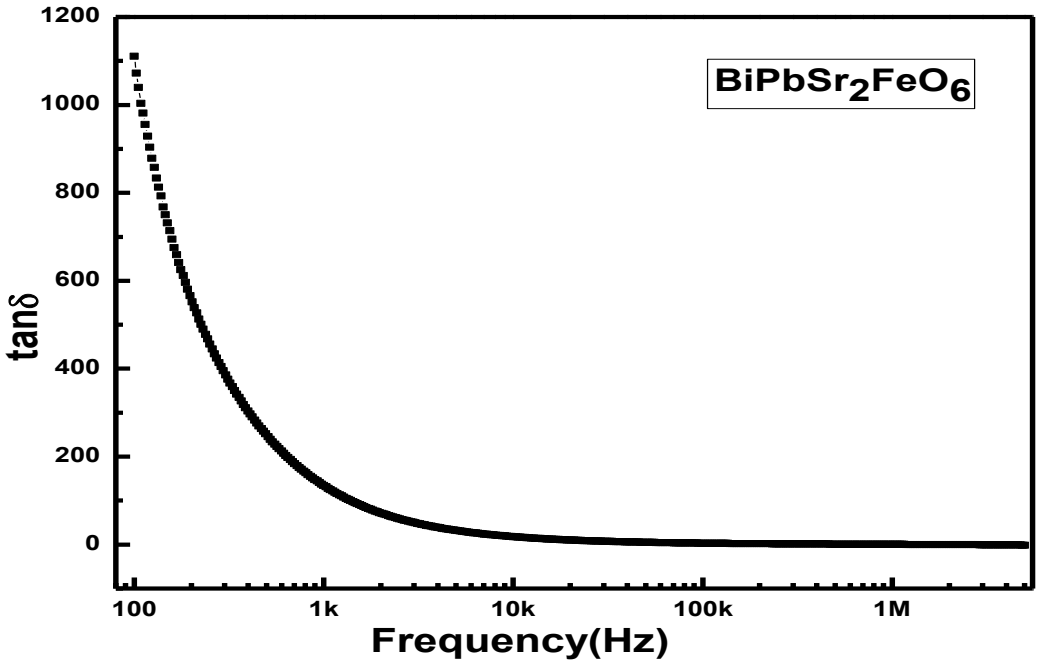

Fig7.8: Variation of dielectric loss of $BiPbSr_2FeO_6$

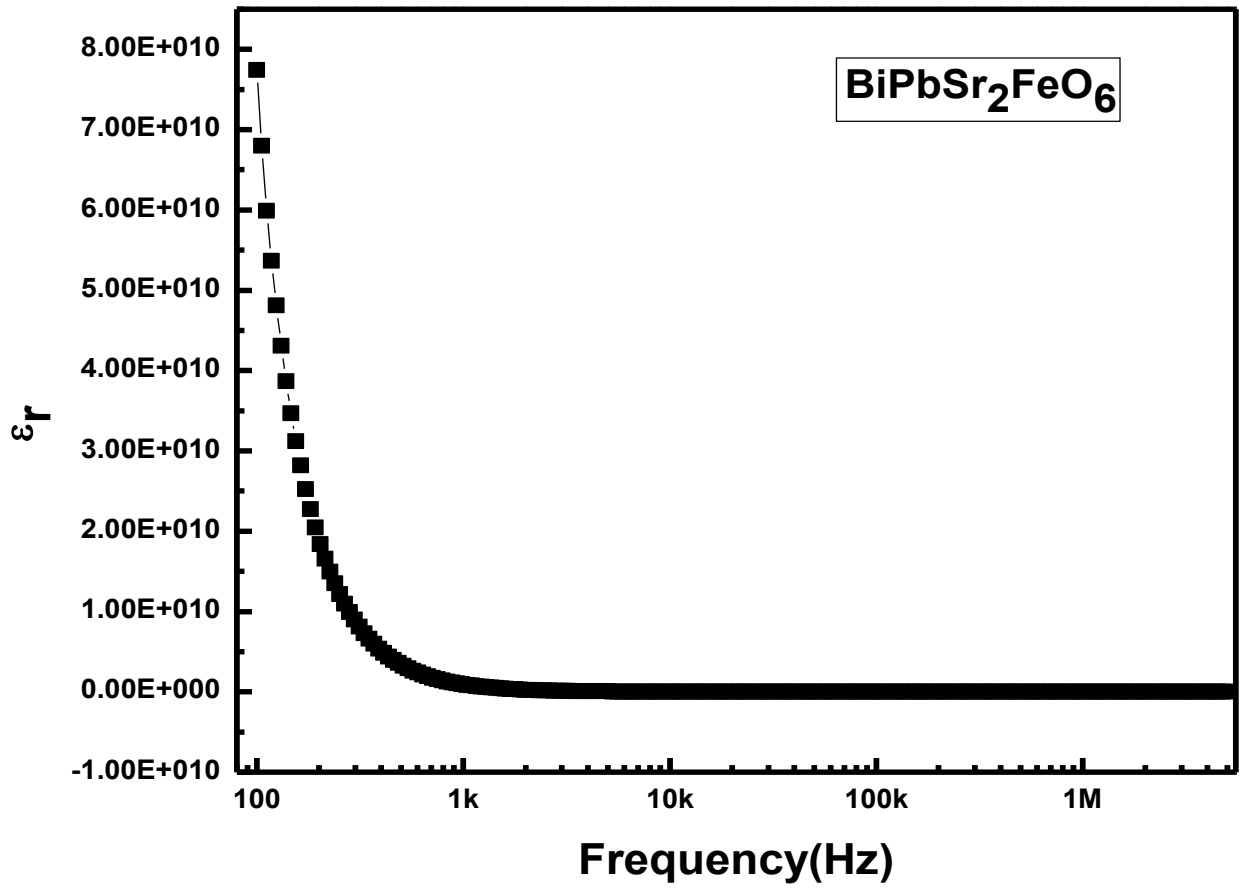

Fig7.9: Variation of dielectric constant of $BiPbSr_2FeO_6$

8 CONCLUSIONS:

From the XRD result the formation of $BiPbSr_2FeO_6$ is conformed and the lattices parameters are also calculate which nearly equals to previous data. FESEM image suggest that the compound is well sintered and grains are uniformly distributed and porosity is visible. Band gap for $BiPbSr_2FeO_6$ is found to be 1.57 eV from the UV-VIS data, it means near to the semiconductor region. Dielectric constant's variation with frequency is also studied, which is decreasing with increasing in frequency, also the loss also decreases with increase in frequency, which conform the role of space charge and orientational polarization in the compound.

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